

# Develop Plan for Analysis of the Effluent from GCM Production

## Fuel Cycle Research & Development

Milestone M4FT-15SN0312045

DOE/NE-Fuel Cycle R&D  
Materials Recovery and Waste Form Development

*Prepared for*  
*U.S. Department of Energy*  
*Office of Nuclear Energy –*  
*Separations Working Group*  
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**Plan:** This milestone is focused on developing a plan for the analysis of the effluent from the Sandia low temperature sintering Bi-Si-Zn oxide glass composite material (GCM) waste form for the long term storage of iodine and its capture materials.

To date, we have been using the thermal gravimetric analysis – differential scanning calorimeter (TGA-DSC; (TA Instruments, New Castle, DE)) with simultaneous mass spectrographic (MS) analysis (Thermo Star™, Pfeiffer Vacuum GmbH, Asslar, Germany) of the off-gas. The GCM is pre-pressed into a pellet, a section is cut off to fit in the TGA pan, and the sample is then heated in flowing air or N<sub>2</sub>, at 5°C/min to 550°C and held at that temperature for 1 hour. It is then cooled to room temperature. During this sintering process, the off gas effluent is collected and recorded by the MS. We have successfully monitored the water, iodine species, nitrates, and organics (in the case of organic iodide in the adsorption studies).

We plan to use these techniques to analyze D<sub>2</sub>O loaded Ag-Mordenite zeolite sample provided by ORNL (R. Jubin, S. Bruffey) in FY15.

In the future case of monitoring off gases such hydrogen isotopes, we plan to utilize these analytical devices:

(1) NMR: solid state and solution nuclear magnetic resonance (NMR) characterization of zeolites and adsorption chemistry will be performed at the SNL NMR facility with houses three instruments dedicated to the characterization of materials in either solid state or solution studies. Both static and MAS NMR solid state probes with variable temperature ranges of -150°C to 150°C are currently available for these experiments.

(2) GC: gas chromatography (GC) with thermal conductivity detection will be performed for the quantitative analysis of static off-gases. Gas samples are typically used, but gases can also be captured from solid or liquid samples for analysis.

(3) GC with ionization detector: Sandia has recently developed method for the quantitative analysis of low levels of water in either gases, liquid, or solids. This is important for off-gas effluent studies as the hydrogen isotopes will probably be associated with offgasing water/steam. This method spans a large dynamic range and is specific to water.

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